Hindawi Publishing Corporation
Journal of Nanomaterials
Volume 2014, Article ID 953717, 8 pages
http://dx.doi.org/10.1155/2014/953717

Research Article

Development of Nanostructured AA3103 by Equal Channel Angular Pressing and Thermal Treatments

C. J. Luis, R. Luri, J. León, I. Puertas, D. Salcedo, and I. Pérez

Mechanical, Energetics and Materials Engineering Department, Manufacturing Engineering Section, Public University of Navarre, Campus Arrosadía s/n, 31006 Pamplona, Spain

Correspondence should be addressed to C. J. Luis; cluis.perez@unavarra.es

Received 4 November 2013; Revised 27 February 2014; Accepted 11 March 2014; Published 7 April 2014

Academic Editor: Edward A. Payzant

This work presents a study related to the achievement of a nanometric structure in AA3103, employing severe plastic deformation processes (SPD), in this case equal channel angular pressing (ECAP). The changes in the mechanical properties and in the microstructure of AA3103 were studied after being processed by ECAP. Subsequently, scanning electron microscopy was used to determine the evolution of the microstructure after different thermal treatments on the material processed by this severe plastic deformation process. Furthermore, a more profound knowledge of the changes in the mechanical properties of this aluminium alloy was obtained. It was demonstrated that with different appropriate combinations of thermal treatments and ECAP processing, it is possible to significantly improve the mechanical properties through obtaining submicrometric grain size structures.

1. Introduction

The equal channel angular extrusion process was first developed by Segal et al. in the former Soviet Union [1, 2]. This process consists in extruding a billet of a polycrystalline material through a die made of two channels with the same cross section that intersect at an angle, 90° in this case, as Figure 1 shows. As the cross-section of the processed materials does not have significant changes, due to the fact that both die channels are manufactured with a similar cross-section size, the ECAP process can be repeated several times over the same billet and, thus, achieve $\varepsilon \gg 1$. [3, 4].

In order to increase aluminium alloy strength, severe plastic deformation processes (SPD) such as the equal channel angular pressing (ECAP) can be used. In this present research work, AA3103 is studied because this alloy cannot be strengthened by thermal treatments.

The ECAP process, in comparison with other severe plastic deformation processes, introduces a more homogeneous deformation and it allows us to obtain parts of larger size. With other conventional plastic deformation processes, such as drawing or rolling, one or more dimensions are modified after each stage of the process. Therefore, in order to get similar values of plastic strain to those obtained by ECAP, the final section or thickness would be so small that this would limit the material processed subsequent application as structural elements.

Grain refinement down to submicrometric or even nanometric level can be achieved by ECAP process. Mechanical properties of the processed material are improved due to this grain size reduction and even superplastic behaviour can be displayed by the material [5]. As the material is processed by ECAP, the existing microstructure is replaced with a new crystalline lattice of submicrometric and/or nanometric structure, due to the introduction of a high density of dislocations in the material. The subgrains of this new lattice have a smaller size and may have a more equiaxial morphology than that of the initial grains. Further thermal treatments allow us to modify the mechanical properties of the processed material to the specific operation requirements.

In spite of the fact that the ECAP process has been widely studied with light alloys, due to the significant improvement in the mechanical properties as a consequence of the microstructure refinement, not many research works related to the aluminium alloys of the 3000 series exist. Along these lines, the work of Bhaumik et al. [6] stands out. In this piece of research work the influence of a stress applied to a billet,

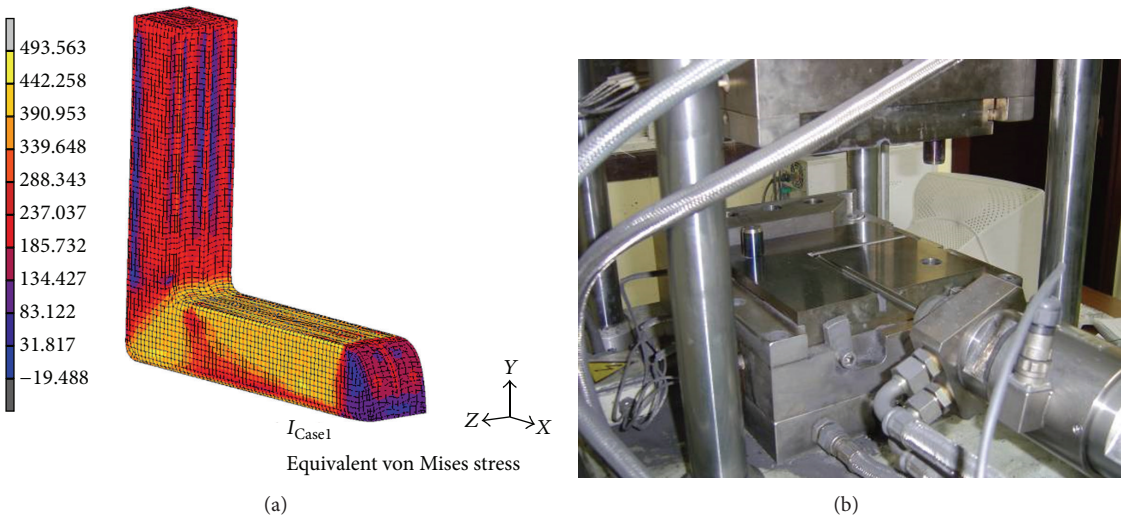


Figure 1: FEM simulation of the ECAP process and ECAP dies.

previously deformed by ECAP using two passages with route Bc, is studied during a thermal treatment of 320°C.

One problem of the severely deformed aluminium alloys is the stability of the microstructure when they are working at temperatures higher than room temperature. As was shown in [7], the higher the annealing temperature, the higher the reduction in the mechanical material properties. An increase in the grain size occurs with the consequent decrease in the mechanical properties such as the yield stress, as can be found in Kang et al. [8]. In this work a sheet of an AA3103 is deformed using a process based on ECAP, called CCSS (continuous confined strip shearing). Roven et al. [9] make a comparison between the yield stress, the tensile strength, and the ductility of aluminium alloys of the 3000, 5000, and 6000 series. The material employed in the tensile tests was previously deformed using the ECAP process and the billets of the 6000 series were also subjected to artificial aging.

In the articles published by Luis Pérez et al. [10, 11] the results achieved for the alloys AA3103 and AA5083 are studied after being processed by ECAP. FEM simulations were performed in order to study the effect of different combinations of angles and geometries for the dies. Besides, different friction conditions and also several operation temperatures were taken into account. Different aspects such as superplasticity, grain refinement, or hardness were related as a function of the number of passes. Therefore, the accumulated plastic strain and its distribution along the cross-section were obtained. In other research works such as Brodova et al. [12], the study is focused on microstructural aspects of an AA3003 processed by ECAP. By employing both optical and scanning electron microscopy along with X-ray diffraction this author highlights the hardening of the sample and the refinement of the structure. This effect is related to the appearance of subgrains delimited by high angle boundaries (HABs). However, there are not many studies that profoundly analyse how the different thermal treatments affect the development of the microstructure and the change in the mechanical properties of the AA3103 processed by ECAP. Therefore, this present research work is intended to improve knowledge of the behaviour of this alloy after being plastically deformed by ECAP and modifying its structure, using different thermal treatments.

2. Experimental Setup

The AA3103 was processed by ECAP at room temperature with an extrusion hydraulic press designed by the Research Group in Materials and Manufacturing Engineering from the Public University of Navarre, shown in Figure 1. The extrusion velocity employed in all the tests was 50 mm/min. The cross-section of the aluminium billets was a square of 9 mm by 9 mm and their length was 80 mm. The lubricant used in order to perform the ECAP process was molybdenum disulphide.

When the same billet is processed by ECAP several times, different routes can be used [13, 14]. In this case route C was selected. This route consists in rotating the billet 180°, around its longitudinal axis, before it is introduced at the entrance channel of the die to perform a new passage. There are different types of geometry dies cited in the bibliography [3, 5, 15, 16]. The most used ones in the past provided an outer radius which was not tangential to the walls of the die [3, 5]. Subsequently, some other improved geometries have been developed, such as those by Luis Pérez [15] and Luri et al. [16]. The die geometries with an intersection angle of 90° with equal fillet radii of 1.95 mm described in [14, 15] have been used in the present study. This geometry is shown in Figure 1.

The material was studied without having been processed by ECAP (N0), processed up to 2 times by ECAP using route C (N2) and up to 4 times using route C (N4). These three states of material were studied by performing tensile tests and microhardness tests.

Each of the hardness (HV) values was assessed from ten random measurements throughout the cross-section of

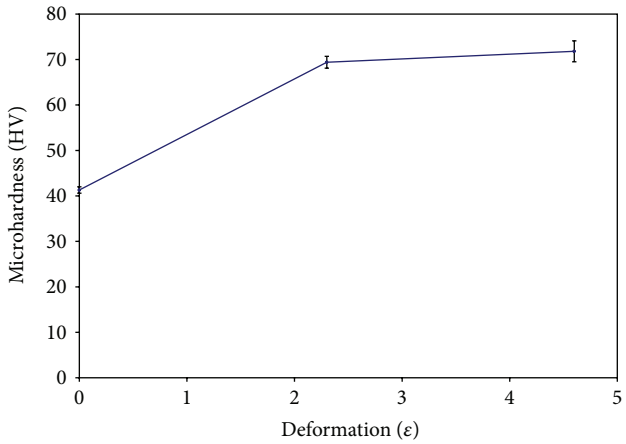


Figure 2: Hardness (HV) of the AA3103 for 1, 2, and 4 ECAP route C passes.

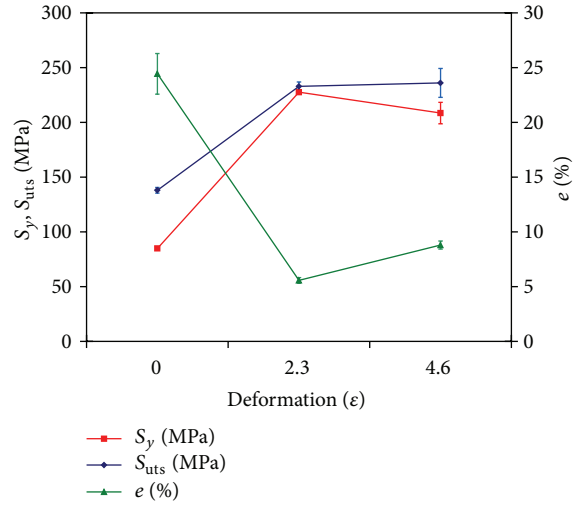


Figure 3: Changes in the mechanical properties: S_y, S_{uts}, and e for the AA3103 as function of the number of ECAP passes.

the samples. The cycle of each indentation is composed of a charging time of 3 s, 10 s of maintaining charge, and 3 s of discharge. The indentation force employed is 3 N.

Tensile tests were conducted for each material state. Three replications were employed for each state. The tensile force in the test was increased with a rate of 100 N/s. The tensile billets were machined from a square ECAP billet with a cross-section of 9 mm by 9 mm and a length of 80 mm. The total length of the tensile billet was of 62 mm. The grip length was 20 mm, the gauge length was 20 mm and the fillet radius between the gauge and the grip zones was 2 mm. All the billets broke in the gauge zone.

In order to study the microstructure of the samples, the samples were cut using a metallographic saw. Subsequently, they were mounted in a nonconductive acrylic resin and they were ground with diamond and colloidal silica as abrasives. To perform the optical microscopy, the Barker electrolytic (2.5% HBF_4) etching was used since it allows us to observe the microstructure under polarized light. In the case of the scanning electron microscopy, the equipment used was a high resolution field emission electronic microscope equipped with detectors of secondary and backscattered electrons along with electron backscattered diffraction (EBSD).

3. Mechanical Properties Analysis

To determine the temperature values for the thermal treatments of this material, a previous study is necessary. This study compares the hardness and searches for the temperature at which recrystallization takes place. In order to do this, the samples were put into the oven once the desired temperature was reached. After one hour of thermal treatment at the established temperature, the hardness (HV) was measured for each sample. Hardness results for the initial state (N0) after two (N2) and four ECAP (N4) passes without thermal treatment are shown in Figure 2. After two ECAP passes, the hardness improvement of 68.1%, as compared to the initial state of the material, does not change significantly if more ECAP passes are applied (at 4 passes the obtained improvement was 71.8%).

Table 1: Hardness measurements obtained for the AA3103.

Deformation (ε)	Temperature (°C)	Time (h)	Hardness (HV)
0	—	—	41.3 ± 0.7
2.3	—	—	69.4 ± 1.3
4.6	—	—	71.8 ± 2.3
2.3	175	1	70.7 ± 2.4
2.3	200	1	70.4 ± 2.0
2.3	225	1	65.0 ± 1.5
2.3	250	1	63.1 ± 2.1
2.3	300	1	53.6 ± 1.0
2.3	350	1	39.0 ± 1.0

The same results are achieved with the mechanical properties obtained from the tensile tests: the yield stress and the tensile strength increase and the elongation at failure decreases after two passes (N2). The properties for four ECAP passes (N4) are similar to those obtained with two passes (N2) due to the effects of plastic strain saturation and work hardening, as Figure 3 shows.

Table 1 shows a summary of the values obtained from the different samples studied, employing Vickers hardness scale.

Figure 4 shows the hardness value for each of the different thermal treatments carried out on the samples in N2 state. The lower line fits to the hardness measurement obtained for the initial state (N0). In the same way, the upper line corresponds to that hardness measurement achieved with two ECAP passes (N2) and without any thermal treatment. After a first peak in which the hardness increases slightly (this could be due to the variability of the hardness tester), a gradual decrease in the hardness appears from 200°C upwards, becoming lower than the initial state (N0) measurement from 350°C upwards.

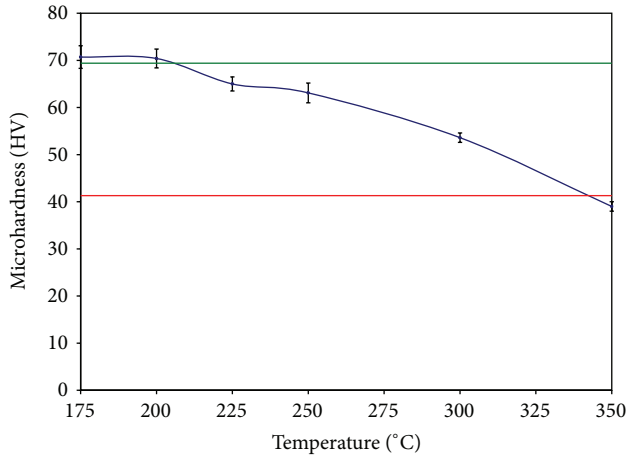


Figure 4: Hardness (HV) for the AA3103 (N2) after the thermal treatments of one hour at different temperatures.

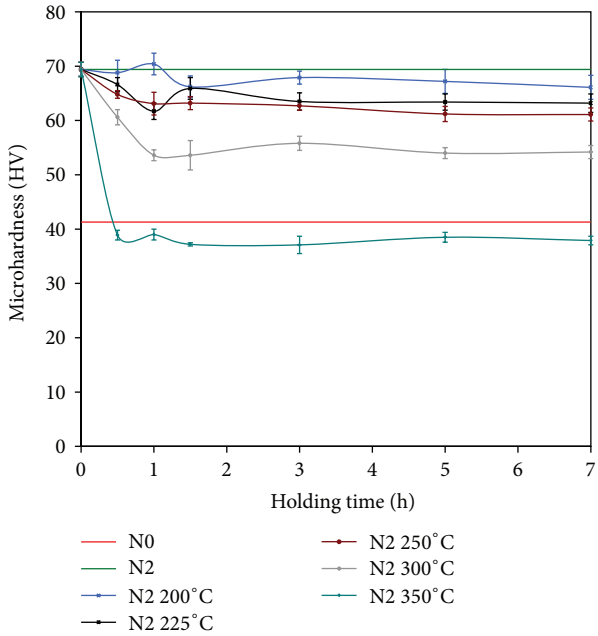


Figure 5: Hardness (HV) for the AA3103 (N2) as function of the holding time at 200°C, 225°C, 250°C, 300°C, and 350°C.

Subsequently, for five of these temperatures (the thermal treatment at 175°C was rejected because the temperature was too low) the samples were thermally treated during different holding times: 0 h, 0.5 h, 1 h, 1.5 h, 3 h, 5 h, and 7 h. Hardness tests along with optical and scanning electron microscopy were carried out on these samples. Figure 5 summarizes the hardness evolution of the treatments carried out.

From the results obtained the thermal treatments at 200°C, 300°C, and 350°C were chosen to carry out the tensile tests. And now, these studies are presented in detail.

3.1. Thermal Treatment at 200°C. For this present study the optimum recovery temperature is considered to be that in

Table 2: Hardness for the AA3103 after different holding times at 200°C.

Deformation (ε)	Temperature (°C)	Time (h)	Hardness (HV)
2.3	200	0	69.4 ± 1.3
2.3	200	0.5	68.8 ± 2.3
2.3	200	1	70.4 ± 2.0
2.3	200	1.5	66.2 ± 2.0
2.3	200	3	67.9 ± 1.2
2.3	200	5	67.2 ± 2.2
2.3	200	7	66.1 ± 2.2

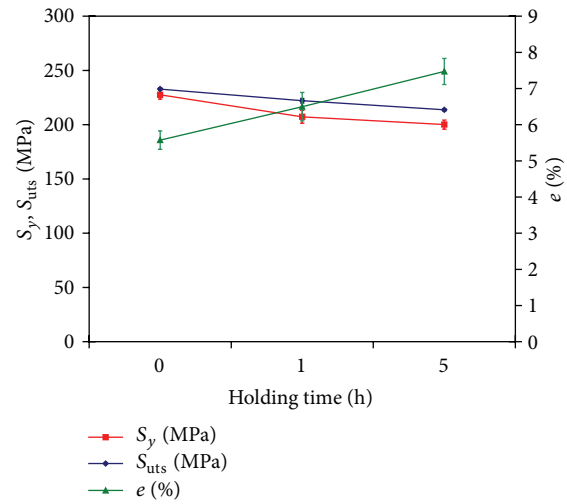


Figure 6: Mechanical properties S_y, S_{uts}, and e for the AA3103 (N2) as function of the holding time of the recovery thermal treatment at 200°C.

which the alloy AA3103 processed by ECAP undergoes a small decrease in its hardness value and, at the same time, it improves its ductility after its specific thermal treatment.

From the previous section the optimum recovery temperature selected was 200°C. Table 2 shows the hardness values measured for each holding time at 200°C as shown in Figure 5.

As can be observed from Table 2, the hardness of the material does not present a significant decrease with the holding time of the treatment, even though for a holding time of 7 h. A small peak appears for the holding time of 1 h. From this time upwards, the hardness begins to decrease slightly until it reaches a stationary value around 66 HV. After these comments, three different holding times, 0 h, 1 h, and 5 h were selected to perform the tensile tests. Figure 6 shows the results obtained.

From Figure 6 it may be stated that both the yield stress (S_y) and the tensile strength stress (S_{uts}) of the material decreases with the holding time of the recovery thermal treatment. Thus, it can be established that for 1 h and 5 h of treatment, a decrease in the yield stress of 8.9% and 12.1% occurs, respectively. For the tensile strength this decrease is around 4.6% and 8.2% respectively. On the other hand, as

Table 3: Hardness for the AA3103 after different holding times at 300°C.

Deformation (ε)	Temperature (°C)	Time (h)	Hardness (HV)
2.3	300	0	69.4 ± 1.3
2.3	300	0.5	60.6 ± 1.4
2.3	300	1	53.6 ± 1.0
2.3	300	1.5	53.6 ± 2.7
2.3	300	3	55.8 ± 1.3
2.3	300	5	54.0 ± 1.0
2.3	300	7	54.2 ± 1.2

Table 4: Hardness for the AA3103 after different holding times at 350°C.

Deformation (ε)	Temperature (°C)	Time (h)	Hardness (HV)
2.3	350	0	69.4 ± 1.3
2.3	350	0.5	38.9 ± 0.9
2.3	350	1	39.0 ± 1.0
2.3	350	1.5	37.2 ± 0.3
2.3	350	3	37.1 ± 1.6
2.3	350	5	38.5 ± 0.9
2.3	350	7	37.9 ± 0.8

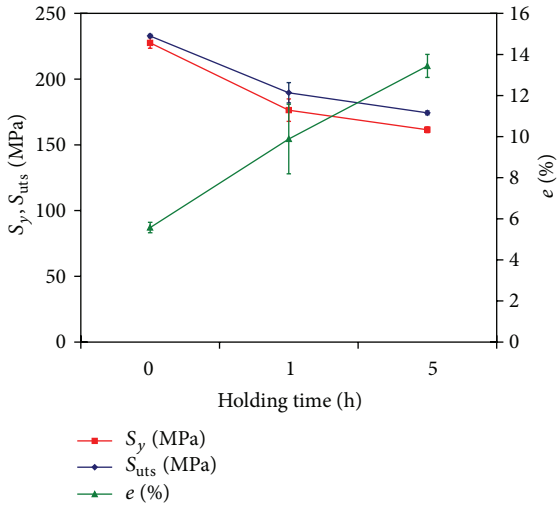


Figure 7: Mechanical properties S_y, S_{uts}, and e for the AA3103 (N2) as function of the holding time of an intermediate thermal treatment at 300°C.

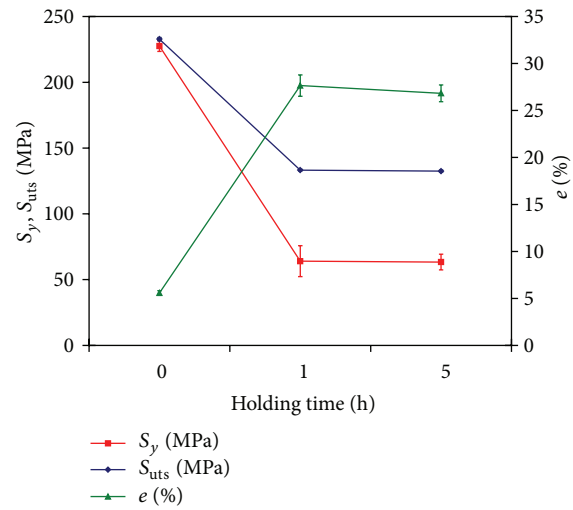


Figure 8: Mechanical properties S_y, S_{uts}, and e for the AA3103 (N2) as function of the holding time of the recrystallization thermal treatment at 350°C.

might be expected, an increase in the ductility is achieved with respect to the billet processed twice (N2) by ECAP. At 1 h of holding time, this increase is 16.6% and around 34% in the case of 5 h.

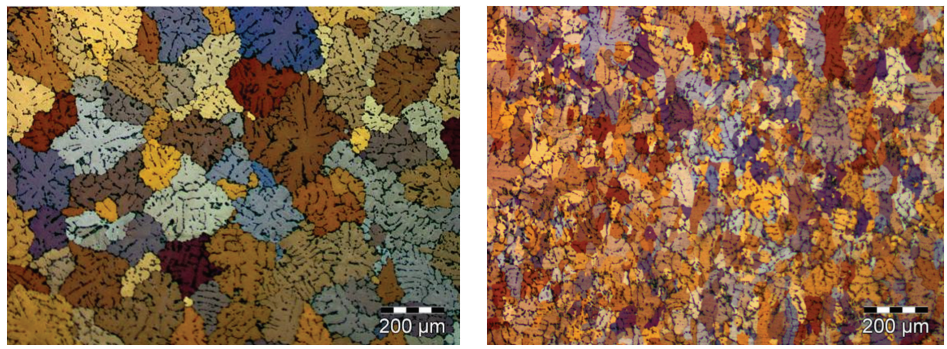
3.2. Thermal Treatment at 300°C. The temperature for this treatment was chosen because it is situated at an intermediate position between the recovery and the recrystallization temperatures (350°C). As will be demonstrated below, the recrystallization does not take place even after 7 h of treatment. This has the aim of obtaining an improvement in the properties without worsening the ductility. Table 3 depicts the hardness values measured for each holding time at 300°C, which are also shown in Figure 5.

Table 3 and Figure 5 show that the hardness decreases in the first hour of treatment a percentage of 23% down to a value of 53.6 HV, where it remains constant or even increases slightly with more hours. After these results, three different holding times of, 0 h, 1 h, and 5 h were selected to perform the tensile tests.

Figure 7 shows the decrease in the yield stress and in the tensile strength of the material when the holding time of the treatment is increased. Thus, the values of decrease in the yield stress are 22.5% and 29% with respect to the holding times of 1 h and 5 h, respectively. Furthermore, the tensile strength decreases by a percentage of 18.6% with respect to the treatment of 1 h and a percentage of 25.1% in the case of 5 h. However, the ductility is improved in the treated material with respect to the material processed twice (N2) by ECAP. At 1 h of holding time this increase is 77.3% and around 141.1% in the case of 5 h.

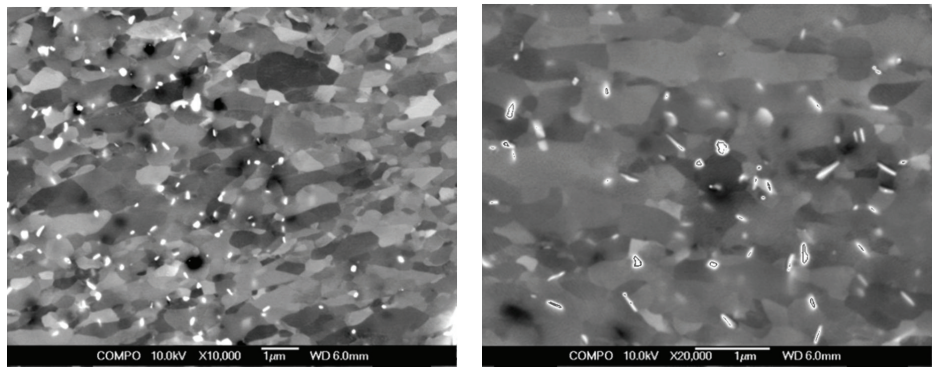
3.3. Thermal Treatment at 350°C. A temperature of 350°C is selected in order to make the recrystallization thermal treatment easier to control. If a higher temperature is employed, the recrystallization process and the decrease in the hardness become too fast. Both factors cause the increase in the grain size of the material. The aim of this treatment is to achieve a decrease in the grain size and to obtain a higher ductility. As is shown in Table 4, the hardness decreases strongly during the first hour of treatment but in a controllable way. After this holding time of 1 h, the hardness values are below than those obtained in the initial state of the material.

As in previous sections, three different holding times of 0 h, 1 h, and 5 h were selected to evaluate the mechanical properties. This was done by performing several tensile tests. The results are shown in Figure 8.



(a) (b)

Figure 9: N0 state (initial material) and N2 with thermal treatment at 350°C during 0.5 h.



(a) (b)

Figure 10: Microstructure after four ECAP passages (N4).

Both the yield stress and the tensile strength of the material decreased considerably. Thus, the percentage of decrease in the yield stress is 71.9% and 72.2% with respect to the holding times of 1 h and 5 h, respectively. The same behaviour is observed for the tensile strength results, where now the percentage of decrease is 42.8% and 43.1%, respectively. On the other hand, an increase in the ductility of the thermally treated material is achieved with respect to the billet processed twice (N2) by ECAP. At 1 h of holding time this increase is 396.1% and around 381.3% in the case of 5 h.

4. Microstructure Analysis

In this section the refinement grain achieved by the accumulation of different levels of plastic strain will be shown for several thermal treatments. N0, N4, and NN2 were selected for this microstructure analysis. The NN2 state is one of the novelties of this work. This state is obtained as follows: the initial material is processed twice by ECAP through route C, then a subsequent recrystallization thermal treatment is applied in order to achieve a refinement in grain size, and finally two new ECAP passes are carried out employing route C. The intermediate thermal treatment is carried out at 350°C during 0.5 h as Figure 9 shows.

If N0 is compared with N4 the grain refinement reached in the material samples is more than 250 times its initial grain size. Furthermore, the size ratio of the microstructure grains has been reduced down to half of its initial value. These two aspects have an impact on the improvement in the mechanical properties and on its homogeneity. Table 5 shows these results.

Table 5: Grain size and grain size ratio for different states of the AA3103.

Deformation state	Grain size (μm)	Grain size ratio
N0 ($\varepsilon = 0$)	≈150	>5
N4 ($\varepsilon = 4.6$)	0.55 ± 0.35	2.41 ± 1.01

Figure 9 shows an optical micrograph of the grain size for the initial material, with a mean value of 150 μm and N2 with thermal treatment at 350°C during 0.5 h (NN2) with a mean value of 50 μm.

In the same way, Figure 10 shows a SEM micrograph of the grain size of the AA3103 after four ECAP passes and rotating the billet 180° between each ECAP pass (route C).

From the EBSD mapping (see Figure 11) the grain misorientation for the sample that has undergone four passes (N4 as worked) is easy to see, because of the big difference in colours, as might have been expected beforehand.

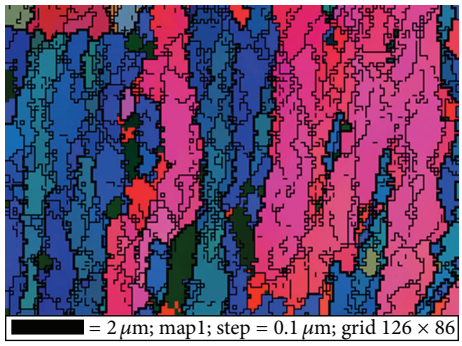
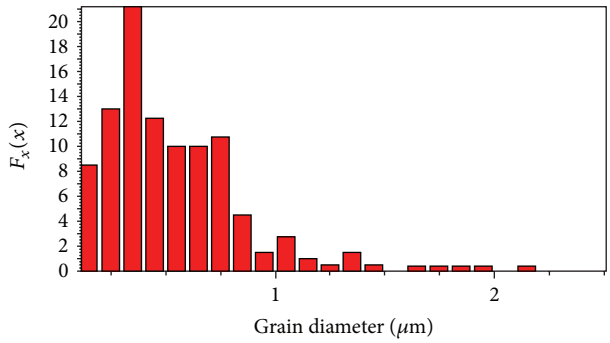
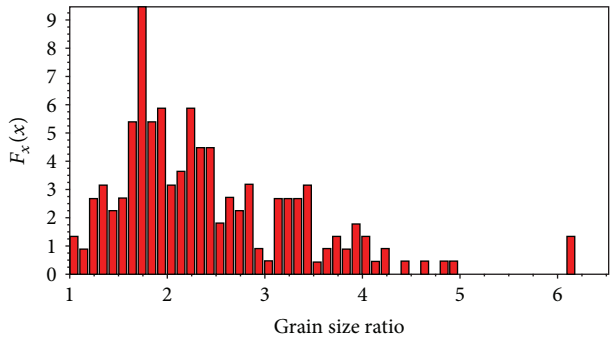


Figure 11: EBSD mapping of N4.



(a)



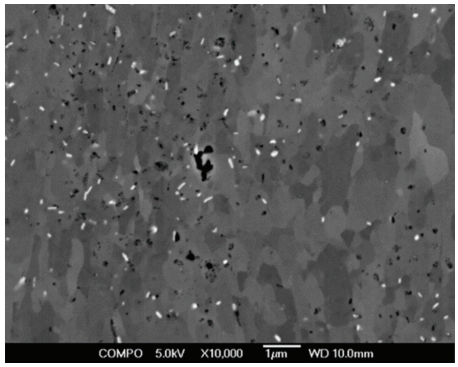
(b)

Figure 12: Distribution function of the grain size and grain size ratio (N4).

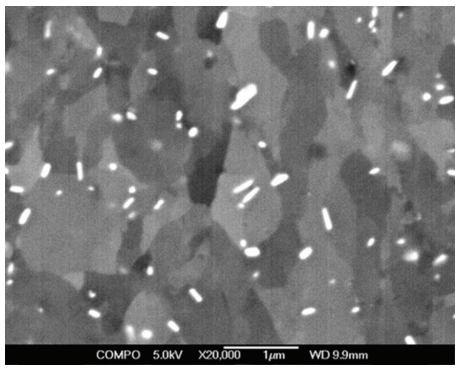
From the EBSD mapping, the histograms shown in Figure 12 are attained. This histogram presents the distribution function of the grain size, which is centred on a value of around 550 nm, and the grain size ratio, whose peaks are in a range between 1.5 and 3.5.

In order to analyse if the grain size value of the NN2 state decreases compared to the N4 state, Figure 13 presents two SEM micrographs showing the grain size of the microstructure. As can be observed, the grain size does not change significantly with respect to N4 (see Figure 10).

The N4 material was deformed up to a plastic deformation of 4.6, where the initial grain size was around 150 μm and a grain size smaller than 1 μm was eventually reached. On the other hand, the NN2 was first processed up to two times



(a)



(b)

Figure 13: Microstructure after processing the material (NN2).

by ECAP introducing a plastic strain of 2.3 and, henceforth, a thermal treatment was used. Then the grain size was modified and the plastic strain disappeared due to the treatment. The grain size after the process was around 50 μm. This new material was then processed twice more by ECAP up to a plastic deformation of 2.3 where, in this case, the initial grain size was around 50 μm and a grain size smaller than 1 μm was eventually reached.

Each material was deformed up to a different plastic deformation level and had different initial grain size. It can be observed from the experiment that the smaller the initial grain size the lower the required plastic strain to be introduced in order to reach the desired grain reduction.

5. Conclusions

In this research work, a study on the variation in the microstructure and in the mechanical properties was carried out for the aluminium alloy AA3103 after being processed by severe plastic deformation (SPD), with equal channel angular pressing process being employed for this purpose.

It was determined that using a thermal treatment at 200°C with a holding time of 1 h or 5 h it is possible to achieve a significant improvement in the mechanical properties. The yield stress can be increased by around 135%. This has a great deal of interest for developing applications where good resistance properties are required. As well as this increase in the tensile strength, it was found that a slight improvement

in the ductility with respect to the twice processed material with route C was also possible. On the other hand, utilising a recovery thermal treatment at 300°C during 5 hours, the yield stress increases very much and with a ductility higher than that of the processed material. Finally, if a recrystallization thermal treatment at 350°C is employed during 1 hour, a very significant improvement in the elongation to failure is achieved with respect to the initial material. This percentage of increase is around 13.2%.

As far as the grain size refinement is concerned, it may be stated that the final size achieved is approximately 500 nm.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

The authors of the present study acknowledge the support given by the Government of Navarre (Project EUROINNOVA NANOCONS-IIM10784.RI1).

References

[1] V. M. Segal, "The method of material preparation for subsequent working," USSR Patent 575892, 1977.

[2] V. M. Segal, V. I. Reznikov, A. E. Drobyshevskiy, and V. I. Kopylov, "Plastic working of metals by simple shear," *Russian Metallurgy*, vol. 1, pp. 99–105, 1981.

[3] R. Z. Valiev, "Structure and mechanical properties of ultrafine-grained metals," *Materials Science and Engineering A*, vol. 234–236, pp. 59–66, 1997.

[4] J. León, C. J. Luis, R. Luri, B. Huarte, and I. Puertas, "Comparative study of the required force for performing Equal Channel Angular Extrusion with routes A and C," *Current Nanoscience*, vol. 3, no. 3, pp. 241–244, 2007.

[5] M. Furukawa, Y. Iwahashi, Z. Horita, M. Nemoto, and T. G. Langdon, "The shearing characteristics associated with equal-channel angular pressing," *Materials Science and Engineering A*, vol. 257, no. 2, pp. 328–332, 1998.

[6] S. Bhaumik, X. Molodova, and G. Gottstein, "Effect of stress on the annealing behavior of severely plastically deformed aluminum alloy 3103," *Materials Science and Engineering A*, vol. 527, no. 21-22, pp. 5826–5830, 2010.

[7] Y. J. Chen, Y. C. Chai, H. J. Roven, S. S. Gireesh, Y. D. Yu, and J. Hjelen, "Microstructure and mechanical properties of Al-xMg alloys processed by room temperature ECAP," *Materials Science and Engineering A*, vol. 545, pp. 139–147, 2012.

[8] H. G. Kang, J. P. Lee, M. Y. Huh, and O. Engler, "Stability against coarsening in ultra-fine grained aluminum alloy AA 3103 sheet fabricated by continuous confined strip shearing," *Materials Science and Engineering A*, vol. 486, no. 1-2, pp. 470–480, 2008.

[9] H. J. Roven, H. Nesboe, J. C. Werenskiold, and T. Seibert, "Mechanical properties of aluminium alloys processed by SPD: comparison of different alloy systems and possible product areas," *Materials Science and Engineering A*, vol. 410-411, pp. 426–429, 2005.

[10] C. J. Luis-Pérez, R. Luri-Irigoyen, and D. Gastón-Ochoa, "Finite element modelling of an Al-Mn alloy by equal channel angular extrusion (ECAE)," *Journal of Materials Processing Technology*, vol. 153-154, no. 1–3, pp. 846–852, 2004.

[11] C. J. Luis Pérez, P. González, and Y. Garcés, "Equal channel angular extrusion in a commercial Al-Mn alloy," *Journal of Materials Processing Technology*, vol. 143-144, pp. 506–511, 2003.

[12] I. G. Brodova, I. G. Shirinkina, O. A. Antonova, E. V. Shorokhov, and I. I. Zhgilev, "Formation of a submicrocrystalline structure upon dynamic deformation of aluminum alloys," *Materials Science and Engineering A*, vol. 503, no. 1-2, pp. 103–105, 2009.

[13] C. J. Luis, Y. Garcés, P. González, and C. Berlanga, "FEM analysis of equal channel angular processes," *Materials and Manufacturing Processes*, vol. 17, no. 2, pp. 223–250, 2002.

[14] Y. W. Tham, M. W. Fu, H. H. Hng, Q. X. Pei, and K. B. Lim, "Microstructure and properties of Al-6061 alloy by equal channel angular extrusion for 16 passes," *Materials and Manufacturing Processes*, vol. 22, no. 7, pp. 819–824, 2007.

[15] C. J. Luis Pérez, "On the correct selection of the channel die in ECAP processes," *Scripta Materialia*, vol. 50, no. 3, pp. 387–393, 2004.

[16] R. Luri, C. J. Luis, J. León, and M. A. Sebastian, "A new configuration for equal channel angular extrusion dies," *Journal of Manufacturing Science and Engineering, Transactions of the ASME*, vol. 128, no. 4, pp. 860–865, 2006.